Synthesis and Characterization of Strontium Titanate

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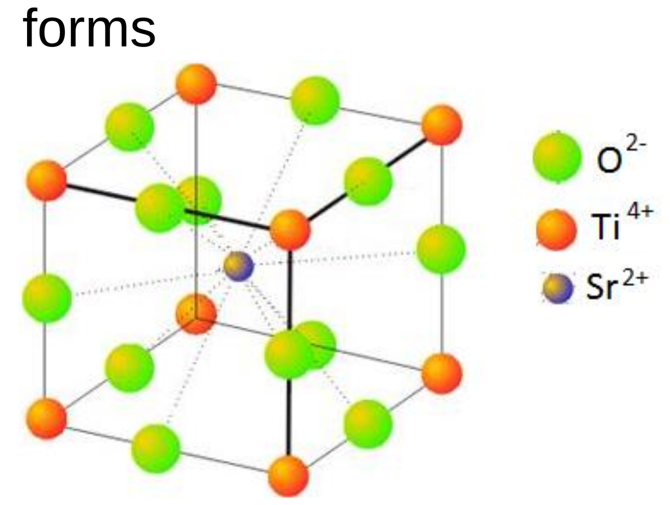
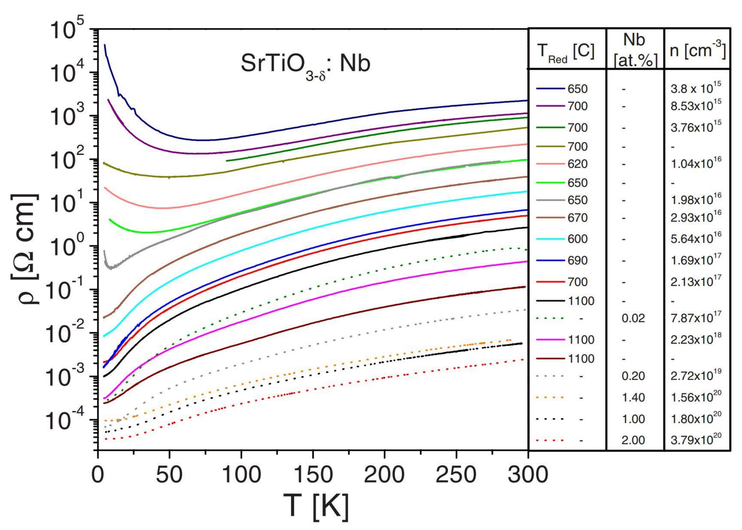
Motivation

- $SrTiO_3$ - Oxide Semiconductor
  - Cubic Perovskite Oxide
  - Lattice Parameter: 3.905 Å
  - Band Gap: 3.2 eV
  - Shows remarkable electronic properties in bulk and thin film forms



Cubic Perovskite structure



Spinelli *et al.*, PRB **81**, 155110 (2010)

- Focus:
  - Investigate influence of deposition parameters on the stoichiometry/defects in thin films of $SrTiO_3$ grown using high pressure oxygen sputtering
  - Study influence of strain and non-stoichiometry on electronic properties

Target Synthesis

- $SrTiO_3$ targets are processed by grinding and reacting $SrCO_3$ and TiO_2 compounds
- Desired quantity of Nb_2O_5, La_2O_3 or other metal oxides can be added for required doping

Grind $SrCO_3$ and TiO_2 for 45 min

↓

React for 72 hours at 1000-1200 ºC with intermediate grinding

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Press target at 40,000 lbs

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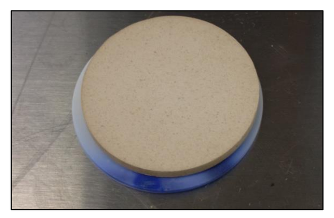
Sinter for 24 hours at 1200 ºC

Powder grinding with mortar and pestle

Mixed powders transferred to alumina crucible

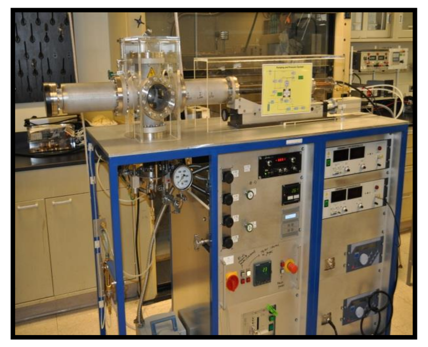
Powders reacted in furnace at 1000-1200 ºC

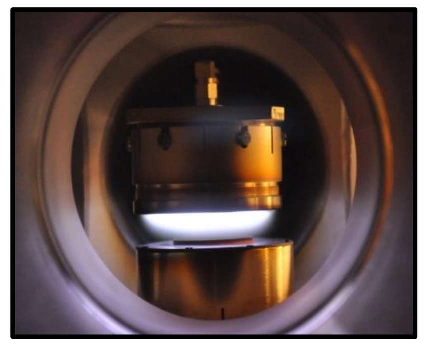


Target pressed and sintered

Thin Film Growth

- 2" $SrTiO_3$ targets
- Substrates:
  - $SrTiO_3$(001) – Growth optimization
  - $LaAlO_3$(001) – Electronic measurements
- T_{dep}: 600 ºC - 900 ºC
- P_{O2}: 1.3 Torr - 2.6 Torr
- R_{dep}: 0.5 Å/min - 2.5 Å/min
- Pre-annealing: 900 ºC, 1.9 Torr
- Natural cooling in vacuum





X-Ray Diffraction

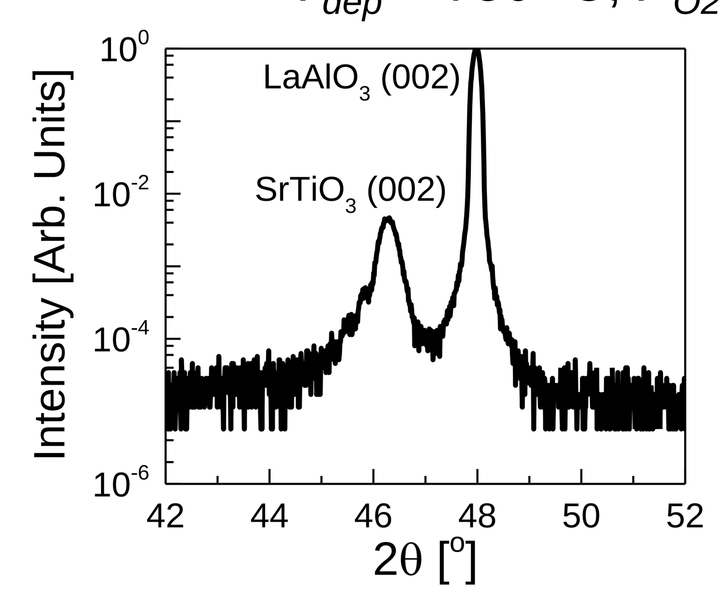
XRD was used to study the structure of films with respect to various deposition parameters and film thicknesses.

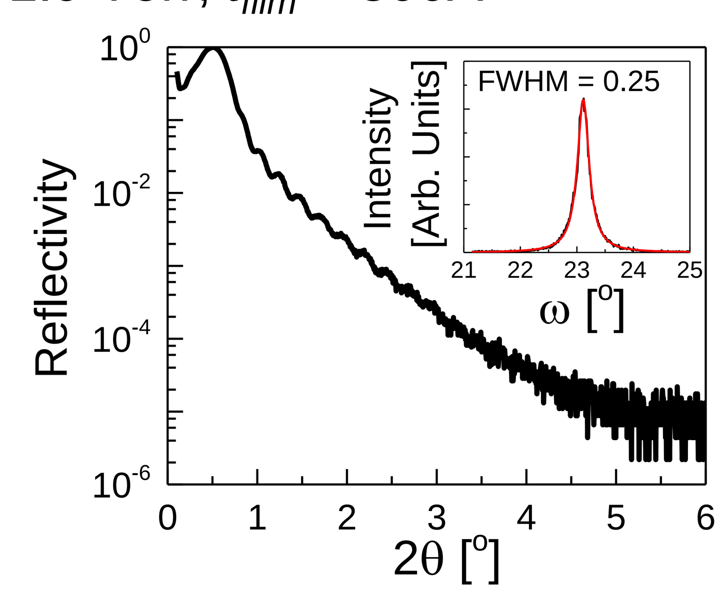
- Wide Angle X-Ray Diffraction (WAXRD)
  - Bragg's law: $n\lambda = 2d\sin\theta$

  Where:
  λ = wavelength of incident X-rays
  d = interplanar spacing
  θ = angle between incident beam and scattering planes
  $n = 1, 2, 3, \ldots$
  - Scherrer equation: $\Lambda = \frac{\lambda}{\beta\cos(\theta)}$

  Where:
  Λ = grain size or correlation length
  β = full-width at half-maximum (in radians)
  θ = Bragg angle
- Grazing Incidence X-Ray Reflectivity (GIXR)
  - Kiessig formula: $\sin^2\theta = \left(\frac{(N+k)\lambda}{2t}\right)^2 + 2\delta$
  - Film thickness t can be determined by frequency of fringes

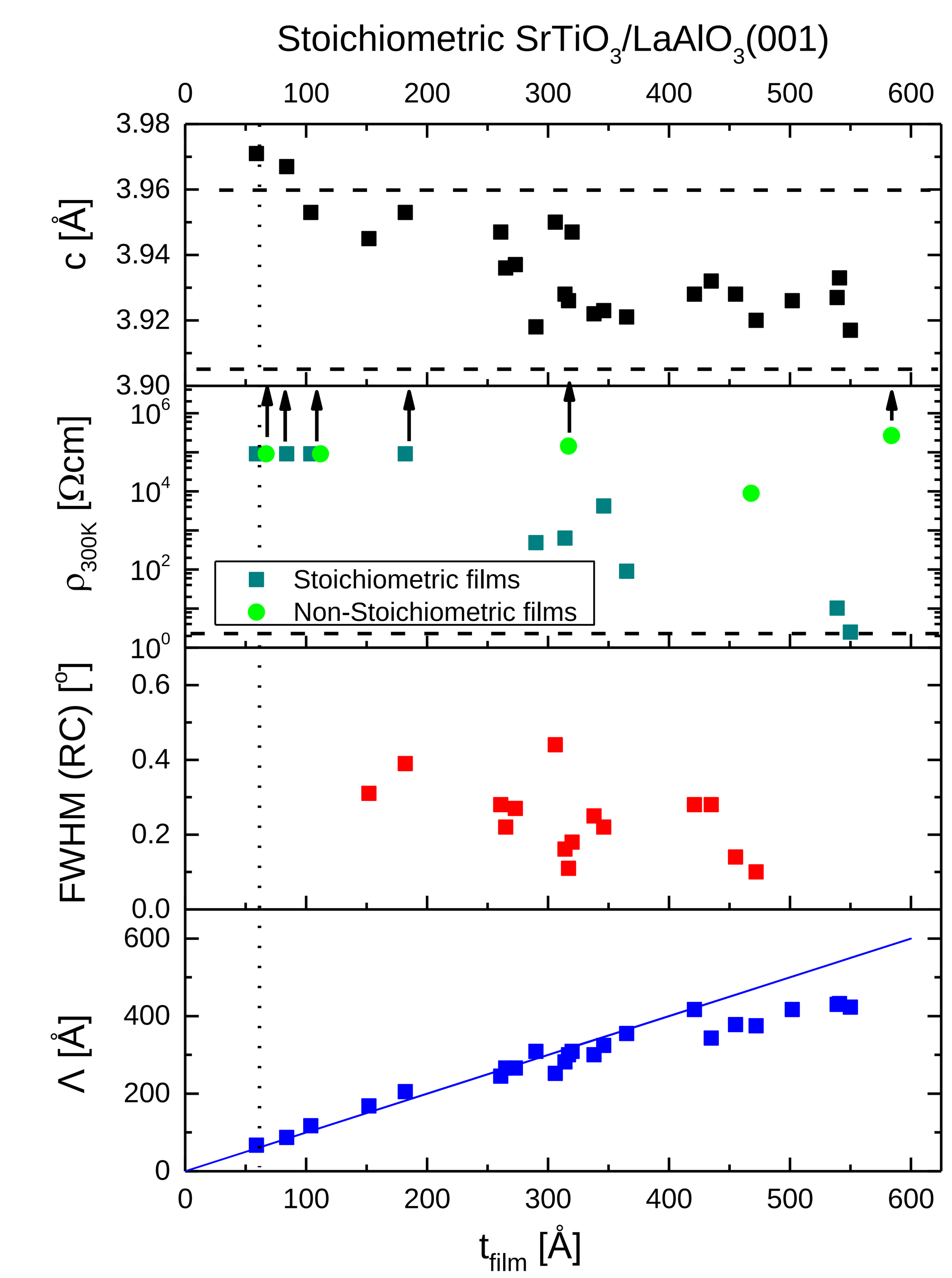
T_{dep} = 750 ºC, P_{O2} = 1.9 Torr, t_{film} = 300Å





Results

- Conditions for Stoichiometric growth: $T \geq 750$ ºC, $P_{O2} \geq 1.9$ Torr, $R_{dep} \leq 1.5$ Å/min
- Stoichiometry determined using WAXRD, GIXR and High-resolution Transmission Electron Microscopy on $SrTiO_3/SrTiO_3$(001)
- Strain relaxation in $SrTiO_3/LaAlO_3$(001) investigated using WAXRD, GIXR and rocking curves
- Room-temperature resistivity measured on films annealed at 900 ºC in vacuum (reduced) to induce oxygen vacancies



Conclusion

- High-pressure oxygen sputtering leads to stoichiometric $SrTiO_3$ films at high growth temperatures and pressures
- Strain-relaxation on $LaAlO_3$(001) substrates occurs in both stoichiometric and non-stoichiometric films at around 300-400 Å as determined by out-of-plane lattice parameter and Scherrer length
- Stoichiometric films have lower resistivities than non-stoichiometric films at all thicknesses
- Thicker stoichiometric films show room-temperature resistivities close to the bulk $SrTiO_3$ crystals reduced at the same conditions indicating low defect densities